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INVESTIGATION OF SOLIDIFICATION OF HIGH STRENGTH STEEL CASTINGS

TECHNICAL REPORT WAL TR 311/49

by

Foundry Section

Metals Processing Division

Department of Metallurgy

Interim Summary Report

D/A Contract DA-19-020-ORD-5443

Boston Ordnance District

OMS CODE 5010.11.842
D/A Project 59332007
Watertown Arsenal
Watertown 72, Mass.

MASSACHUSETTS INSTITUTE OF TECHNOLOGY
Cambridge, Mass.

Cast Steel
Microsegregation
Solidification
Homogenization

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ABSTRACT

Results are presented of the first six months of a continuing research program on solidification of high strength cast steel. Exploratory experiments were conducted and apparatus assembled to permit solidification at controlled rates of cooling of small samples of steel or lower melting point materials; these experiments can be carried out under controlled atmosphere. The apparatus permits solidifying samples at rates from approximately 500°F. per minute to rates less than 0.005°F. per minute.

Dendrite arm spacing in steel is shown to be markedly dependent on both cooling rate and alloy analysis. For example, in a low alloy steel, spacing is 900 microns at a cooling rate of 0.43°F. per minute during solidification; spacing is 260 microns at a cooling rate of 19.2°F. per minute. In a more highly alloyed nickel-chromium steel, dendrite spacing is less than 85 microns over a comparable range of cooling rates.

Dendrite arm spacing is the primary structural factor in castings and ingots affecting efficiency of homogenizing treatments. Analyses included herein indicate that commercial heat treatments for low alloy steel castings and ingots result in little or no homogenizatior of most elements (except carbon).

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I. INTRODUCTION AND LITERATURE SURVEY

A. General

This interim report summarizes results of research conducted at M.I.T. on solidification of steel during the six-month period June 9, 1961, to December 8, 1961. The overall program, of which the research described herein forms an initial phase, is aimed at determining in detail the nature of, and factors influencing, microheterogeneities which result from solidification. These microheterogeneities include (1) microsegregation, (2) microporosity, and (3) precipitation of interdendritic second phases (inclusions, etc.). Each of them is known or suspected to have significant effects on mechanical properties of cast and wrought steel.

Research work reported builds on the foundation of an earlier extended program conducted at M.I.T. on high strength steel castings, sponsored primarily by Army Ordnance through Watertown Arsenal. That program was carried out during the period 1 July, 1957, to 1 July, 1960; its major emphasis was on examining (1) different methods of obtaining directional solidification in cast steel, and (2) effects of solidification variables on structure, segregation, and properties of cast steel. Alloys AISI 4330 and AISI 4340 were studied, in the air and vacuum melted conditions.[1-7]

Results showed that when careful engineering controls is exercised so as to minimize micro-heterogeneities resulting during solidification, remarkably high mechanical properties can be obtained in cast low alloy steel. For example, in one study of effects on properties of inclusions, melting practice, and solidification techniques, ultimate tensile strengths as high as 317,000 psi were obtained in the cast and heat treated material. In heats corresponding to AISI 4340 analysis, tensile strength was 250,000 to 260,000 psi with reduction in area 37 to 40 percent and elongation 10.0 to 12.5 percent.[7]

In another study on "unidirectional" solidification, properties in a 4-inch diameter casting averaged 290,000 psi ultimate tensile strength, 230,000 psi yield strength, and 17 percent reduction in area. The structure of this casting was entirely columnar and properties perpendicular to the grain orientation were essentially the same as those parallel the grains.[6]

Research described above is finding application in commercial foundries and ingot shops. One example is that Watertown Arsenal has applied the results to production of heavy steel castings, including breech rings. Objectives in the program have been to (1) improve the soundness and mechanical properties of the breech rings, and (2) reduce their cost of production. Both objectives were felt to be possible since if a casting is solidified essentially "unidirectionally" considerable savings may be made in risering and padding, and in subsequent machining to remove these risers and pads.[8]

Results of the program to date have been dramatically successful. One breech ring selected for study (M-68, 105 millimeter) has been produced with fully columnar cast structures, and properties obtained throughout the 7-inch thick ring exceed those one would normally find in a well-fed steel casting (of the same alloy) 1-inch thick.[8]

The techniques are being instituted in production and substantial improvements in economy and quality are anticipated; a saving of over several hundred thousand dollars is projected over the course of the breech ring production.

Fundamental aspects of research to dateat M.I.T. on high strength steel castings have been concerned to a large extent with microporosity and its influence on properties. Research on microsegregation, inclusions, etc. has received less emphasis. These latter factors, however, are also important in determining properties of both cast and wrought materials; recent studies, for example, indicate the probably significant effect of microsegregation on properties of low alloy steels.[9-11] A major part of the study reported herein is concerned with dendritic structure and microsegregation in steel.

B. Dendritic Structure

Cast steels, like most other alloys, solidify with a "pine-tree like" (dendritic) grain structure.* When thermal gradients are sufficiently steep, the dendrites which form may be aligned parallel to the heat flow direction; in this case, a "columnar" macrostructure results. When thermal gradients are relatively small and adequate nuclei are present for crystallization, dendrites form and grow at random within the solidifying mass; in this case, the final grain structure is composed of randomly oriented, equiaxed dendrites (i.e., grains).

Hence, thermal and other solidification variables have a marked influence on the as-cast grain shape and size. Solidification variables also affect the internal structure of individual grains; that is, the number and spacing of dendrite arms. The fineness of the internal dendrite structure is of great practical importance since (1) the bulk of microsegregated constituents are to be found between dendrite arms rather than only at grain boundaries, (2) microsegregation is extremely difficult to homogenize in cast steels at ordinary times and temperatures (as will be shown later), and (3) microsegregation may have a profound influence on the properties of cast and wrought materials.

---

* Pine-tree like dendrites form and grow during solidification. Each dendrite grows to form a grain; hence, each grain is composed of one dendrite.

A variety of studies on ferrous and non-ferrous metals have shown dendrite arm spacing to be markedly dependent on cooling rate, and, in most cases, to be directly proportional to the square root of the solidification time (inversely proportional to the square root of the cooling rate); Alexander and Rhines,[12] Michael and Bever,[13] Reed,[14] and Brown and Adams[15] show this to be true for various non-ferrous metals. Alexander and Rhines, among others, show that solidification time (or cooling rate) is the only significant variable for a given alloy analysis; dendrite arm spacing is unrelated to grain size or shape. One theoretical analysis has been developed showing the effect of cooling rate and other parameters on dendrite arm spacing,[15] and there is a great need for more detailed fundamental study in this area.

Studies of effects of cooling rates on dendrite arm spacing in steel have been more limited. Sauveur and Chou[16] and many others show that dendrite spacing in steel increases with decreasing cooling rate. Work at M.I.T.[3,6] on 4340 alloy shows that this spacing is inversely proportional to the square root of the cooling rate during solidification. It is, therefore, to be concluded that factors governing dendritic growth in steel are similar to those in the non-ferrous alloys described above.

Some attention has been paid to the effect of alloy analysis on dendrite arm spacing. Investigators have found dendrite arm spacing in aluminum may be either increased or decreased by increasing solute content[12,13,15]. Sauveur and Chou[16] indicated that the normal impurities found in commercial steels result in somewhat coarser dendrites than those of purer laboratory heats. Sauveur and Reed[17] found coarsening as a result of nickel additions to cast steel. Martin and Martin,[18] adding a variety of elements including chromium, nickel, and molybdenum to cast steel, also found a coarsening of dendrite arm spacing.

The reason, or reasons, why some added elements should coarsen dendrite arm spacing and others should refine these spacings is not fully clear, but in view of the probable mechanism of dendrite growth, the explanation will most likely be found in terms of phase diagram considerations and those of mass transport during freezing; in some cases, alloy elements may also alter solidification range or thermal conductivity of the solid metal sufficiently that freezing rate is also altered. Specifically, the variables which alloy additions might affect and which would be expected to have an influence on dendrite arm spacing would be (1) shape and position of the liquidus and solidus lines of the alloy, (2) amount of solute to be transported during solidification, (3) rate of diffusion of solute in the liquid or the solid, and (4) freezing rate of the alloy.

C. Microsegregation

The term "microsegregation" is often used indiscriminately to apply to two separate but related phenomena. These are (1) concentration gradients within dendrite arms; i.e., "coring", and (2) precipitation of non-equilibrium phases between dendrite arms and between grains. This latter type of micro-segregation results when the inter-dendritic solute content near the end of freezing reaches such a high value that a new phase precipitates.

The mechanism of formation of microsegregation has been extensively discussed. It results primarily because of the vastly different rates of diffusion of solute in liquid as compared with solid - and it can be amazingly severe. For example, it is not uncommon for the last interdendritic liquid which freezes in a casting to have as much as thirty times as much solute content as the first liquid to freeze. Hence, the spaces between dendrite arms and between dendrites may contain thirty or more times as much solute as the central portions of dendrites which freeze first.

The extent of microsegregation has been the object f considerable attention recently, in part because of the availability of new tools for quantitative or semi-quantitative analysis, and in part because of a growing realization of the effects of microsegregation on properties. One modern technique for studying segregation consists of autoradiography.[13,19-26] In this instance, a specimen of cast metal is subjected to neutron bombardment for a predetermined length of time. Then, assuming the solute has a different (preferably longer) half-life than the solvent, extent and distribution of microsegregation can be measured by standard radiographic techniques. An alternative method is to prepare a casting using a radioactive isotope of the solute in the alloy.

Another method which can be at least semi-quantitative is that of microradiography. In this case, the analysis depends on the solute element having a substantially different coefficient of absorption for X-rays of the wave lengths used than does the solvent.[27,28]

A third method of determing microsegregation is by use of an electron beam microprobe, in which chemical analysis of an extremely fine portion of the sample (as little as 1.5 microns in diameter) is measured.[10,20,24,29,30] Thus, variations in chemical compositions across dendrite arms can be directly measured (since dendrite arms in castings and ingots are generally the order of 10 to 1000 microns thick). One limitation of the microprobe is that those now in use can only detect elements whose atomic number is higher than 11. This, however, includes most of the elements of interest in cast steel, except carbon.

D. Experimental Studies on Microsegregation

A variety of different investigations have been conducted recently on the tendency of various elements to segregate in solidifying steel. These investigations have been conducted using autoradiography or electron microprobe analysis as described above. Efforts to date have been primarily directed to specific commercial alloys and results do not yet provide the fundamental information needed to rationalize fully the factors influencing segregation tendency. Among results of some of the work obtained by use of autoradiography are the following:

Arsenic and phosphorous have similar segregating tendencies (in low alloy cast steels) with arsenic segregation more difficult to reduce by subsequent homogenizing treatments.[23] In one study, severity of microsegregation of various alloy elements (in several different steels) was found to decrease in the following order: molybdenum, manganese, chromium, and nickel, with silicon probably being between manganese and nickel.[24] Other investigators, studying a 1.5 percent nickel-chromium-molybdenum steel, found segregating tendency to decrease in the order molybdenum, chromium, manganese, and nickel.[30] Generally, those elements for which macrosegregation is most severe are also those elements that tend to microsegregate most severely.[24] Regarding other elements, another investigation has shown that in a 0.4 percent carbon steel, cobalt does not segregate at all, molybdenum segregates slightly, and manganese, copper and tungsten segregate appreciably but much less than arsenic.[25]

A series of microprobe studies have been summarized, showing the influence of various concentrations of impurities and alloying elements on segregation in steel.[20,24] The results are, however, too diverse to allow generalization. More recently, also using the electron microprob studies were conducted on a series of binary ferrous alloys. In this wo "segregation coefficient"* for different elements in cast steel was shown to vary from about 1 to about 40 depending upon the element.[20]

---

* "Segregation coefficient" is defined as the ratio of the maximum concentration of alloy element in the interdendritic area to the minimum concentration of alloy at the dendrite axis.

One interesting aspect of microsegregation in solidifying steel concerns the interaction of various elements of their segregating tendency. For example, one study[25] on binary alloys of carbon-free iron showed by autoradiography no detectable segregation of cobalt, manganese, arsenic and other elements, whereas the same solutes present in 0.4 percent carbon steel segregate severely. This result is assumed to be related to the fact that carbon probably appreciably increases the solidification interval. It is of interest that in another study, carbon was shown to reduce considerably the tendency of arsenic and phosphorous to segregate.[20]

Another type of interaction of interest is the one between sulfur and manganese. Manganese segregation is reduced by increasing sulfur content (in the range .008 percent to .150 percent). In regions adjacent sulfide inclusions, the manganese segregation even reverses itself (in thse regions manganese content may be as low as 0.40 percent in a steel of nominal composition 1.40 percent manganese, .150 percent sulfur). The amount of manganese itself present has little or no effect on segregation coefficient which is 1.90 $\pm$ .05 for alloys from 0.70 percent to 4.05 percent manganese (in steel containing .008 percent sulfur).[24]

Using the microprobe, one group of investigators showed the degree of segregation in equiaxed grains is comparable with that in columnar grains (for low alloy steel);[10] another study indicated that microsegregation of chromium, nickel, manganese, and molybdenum is more severe in the equiaxed than the columnar grains.[30] Further work is particularly desirable in this area to establish relationships between microsegregation, grain structure, and feeding parameters during solidification.

Microsegregation in low alloy steel is not eliminated by ordinary commercial homogenizing treatments; a few hours at 2200°F. has little or no effect on distribution of alloy or on the degree of segregation.[10] In fact, such treatments do not apparently homogenize at all elements such as chromium, nickel, molybdenum, etc. in most castings and ingots.

One improtant factor influencing the ease with which a cast structure may be homogenized is the coarseness of the composition gradations within the structure, that is, the dendrite arm spacing. Generally, it is to be expected that the finer the dendrite arm spacing the more rapid the homogenization that can be obtained. This is clearly true as can be seen by comparing results on solution treating welded structures in aluminum - 4.5 percent copper alloy[15] with earlier work on solutionizing the same alloy in cast form.[31] However, in some alloys at least, even though solidification may be as rapid as that obtained in weld deposits, homogenization is extremely difficult.[32] This is in spite of the very fine dendrite arm spacing obtained in weldments.

Very little work has been done to date to determine the effect of cooling rate on microsegregation. Clearly, decreasing cooling rate increases the dendrite arm spacing and hence coarsens the composition gradations. However, the effect of cooling rate on the ratio of maximum to minimum concentration of alloy element after solidification (segregation coefficient) has not been fully delineated. It is to be expected that decreasing cooling rate would have either essentially no effect on segregation coefficient or would decrease this coefficient somewhat. Most studies in the past have indicated the latter is true, although many of these are open to some question since samples that were solidified slowly were also generally cooled more slowly after solidification. Hence, added time was allowed for homogenization. However, in a recent study at M.I.T. on a non-ferrous alloy, the alloy was quenched immediately after solidification; segregation coefficient was nonetheless found to decrease slightly with decreasing cooling rate.[33]

In recent studies on nickel-chrome-molybdenum steels, intensity of microsegregation apparently increased as cooling rate increased.[34,35] Similar behavior has been found for welds, though in a range of much greater cooling rate.[37]

Still another aspect of segregation concerns the banded structure in wrought steels resulting from elongation of dendrites and dendritic segregation in the rolling direction. In one study on a plain carbon steel (0.42 percent carbon, 0.21 percent phosphorous), it was shown that the segregation coefficient of phosphorous in the as-cast material is 4; this can be reduced to 1.5 after extensive heat treatment (64 hours at approximately 2200°F). On the other hand, reduction by working (up to

70 to 1) permits much more rapid homogenization due to the reduction in effective dendrite arm spacing obtained by the working operation.[22]

In a study on effect of working on segregation in 52100 alloy (1.5 percent chromium, 1.0 percent carbon, 0.4 percent manganese) now being conducted at M.I.T. results include the following:

Substantial microsegregation remains (in banded array) in the wrought alloy after reductions in area of as much as 2750 to 1. Even after heat treatments as high as 2100°F during the processing of these ingots, the ratio between maximum and minimum compositions across the banded structure is found to be very little less than the original segregation coefficient in the cast structure. Continuing examination is being given to this problem in our laboratory on low alloy steel such as AISI 4340, as well as on 52100.

II. EXPERIMENTAL AND ANALYTICAL STUDIES

A. Initial Solidification Studies

A series of exploratory solidification experiments were undertaken on an alloy containing 0.2 percent carbon and 2.00 percent nickel. In one such experiment a relatively large ingot (8 pounds) was solidified in a pre-heated gas fired furnace. In this experiment, the mold was first made using rammed mullite refractory; the mold cavity was 2-3/4 inches diameter, by 12 inches high (with a gating arrangement to allow filling with molten metal). This was preheated to 2100°F in a gas-fired furnace and metal was poured directly into the mold while it rested in the hot furnace. Cooling rate was measured by a thermocouple adjacent to the mold-metal interface; after cooling, the ingot was sectioned, etched and structure examined. In this sample, average cooling rate during the solidification interval was 19.2°F per minute; measured dendrite arm spacing was 260 microns.

In view of the relatively rapid rate of cooling obtained above (in spite of the special precautions taken), it was decided that substantially different experimental techniques would be necessary to study solidification of more slowly cooled castings. Hence, an experiment was conducted in which a very small sample (1 inch diameter, 35 grams) was melted in a heated resistance furnace and cooled at a very slow rate under an argon atmosphere. Control equipment on the furnace employed permitted control within ±1°C, and cooling was manually controlled by decreasing the temperature controller gradually. By this method, a cooling rate of .43°F per minute was obtained. (Hence, cooling rate was approximately 1/50th that in the previous experiment.) In this specimen, dendrite arm spacing was 900 microns. These data are summarized in Figure 1, together with other data to be discussed in Part II of this report.

B. Studies on Iron-Nickel-Chromium Alloy

Alloy Preparation and Apparatus

In view of the successful exploratory experiment using the resistance heated furnace described above, a more suitable furnace was assembled and more accurate temperature controller obtained (±1/8°F accuracy). In addition, the controller employed could be, and was, used to automatically control rate of cooling in a manner described below.

The alloy chosen was an iron-nickel-chromium alloy, similar to 309 or 309S austenitic stainless steel. The relatively low melting point of this steel alloy was a major factor influencing its choice, and results obtained using this relatively "rich" alloy proved to be of considerable interest.

Small ingots, 1 inch in diameter by 1 inch high were cut from 8-inch long bars which had been previously prepared by induction melting and sand casting. The initial charge was made of electrolytic iron, nickel, and low carbon ferro-chrome. Final chemical analysis was 25% Cr, 14.6% Ni, .026% C, .008% S, .008% P, balance Fe. The alloy has a liquidus temperature of about 2630°F and a solidus of approximately 2545°F (as measured in this investigation).

Before each run, the charge to be used for a melt was carefully cleaned, placed in an alumina crucible, and inserted in the furnace for melting and subsequent solidification. The crucible was 1" diameter x 1" high (inside dimensions). The furnace was a horizontal tube furnace with silicon carbide heating elements. A 1-1/2 inch diameter zirconia tube was placed within the heating elements; this comprised the furnace walls and permitted maintaining an inert atmosphere around the metal during melting and subsequent solidification. Maximum operating temperature of the furnace was approximately 2750°F.

Temperature control was by platinum - platinum 10 percent rhodium thermocouple in a fused silica tube; the thermocouple bead was placed adjacent but not in the molten metal (to avoid contamination or nucleation of the molten metal). A current-adjusting temperature controller was employed.

Cooling rate control was obtained by employing a differential thermocouple placed inside the resistance furnace. One junction of the differential thermocouple was embedded in a block of ceramic; the other was placed just outside the block of ceramic (but both were within the hot zone of the furnace itself). By accurately controlling the temperature <u>difference</u> between these two junctions, quite wide variations in cooling rate could be obtained.

Experimental Procedure

After placing the charge material inside the furnace and establishing a positive pressure of inert gas (helium), the furnace temperature was gradually raised to 2700°F and maintained at this temperature for one hour, a time sufficient for melting the sample. Then, the furnace was cooled slowly, cooling rate being controlled by the differential thermocouple described above. At the end of the cooling cycle, usually at some temperature slightly below the estimated solidus, the sample was quickly removed from the furnace and quenched (generally in water).

The quenched samples were cut along their longitudinal axes, mounted and polished for metallographic examination. They were first examined for inclusions at 100X. Dendritic structure was then revealed by swabbing for about 10 seconds with an etch prepared according to the following formula: nitric acid, 5 parts; hydrochloric acid, 2 parts.

Results and Discussion

Samples cooled at five different rates were examined and are reported herein; these rates ranged from that obtained by water cooling to a rate of only 1°F per minute. The fastest cooling rate (water quench) was obtained by melting a sample in the furnace, allowing it to partially solidify and then quenching. Dendrite arm spacing in that portion of the sample that was liquid when quenched was 26 microns, Figure 2.

Partial solidification and then water quenching should prove a useful tool to delineate the form and distribution of the first solid to freeze in steel castings. Figure 3 illustrates the boundary of a dendrite arm that was growing in the above sample while it was slowly cooled and before it was quenched. Apparently, the vastly increased cooling rate resulting from the water quench caused the coarse arm to quickly project little "cells" or small dendrite arms and these then broke down into the structure shown.

The next slower cooling rate studied was 720°F per minute, Figure 4. This is the approximate cooling rate obtained when the alloy is poured into a cold crucible (of the size used) or cold sand mold cavity. Dendrite arm spacing at this cooling rate is 52 microns.

Slower cooling rates were obtained by carefully controlling cooling as described above. Those examined were 75, 1.15, and 1.00°F per minute. Dendrite arm spacings were, respectively, 52, 70, and 84 microns. Structures are shown in Figures 5, 6 and 7.

Results of measurements of dendrite arm spacings are given in Table I and plotted in Figure 1. They are plotted as dendrite arm spacing versus the square root of the reciprocal of cooling rate. Results are compared with those described in Part A of this section, and from earlier, similar investigations. Conclusions of major interest to be derived from Figure 1 are that (1) the dendrite arm spacing of the iron-nickel-chromium alloy at a given cooling rate is substantially smaller than that of the other, more dilute, alloys studied, and (2) the slope of the curve is also substantially less; that is, rate of cooling has much less effect on dendrite arm spacing in this alloy than in alloys which contain less solute. The significance of the above is that it lends promise to the hope that substantial reductions in dendrite arm spacing can be effected in practice through addition of alloying elements. Through this reduction in dendrite arm spacing, effective homogenization treatments might become possible and with them improved mechanical properties.

Because of variations in amount and type of inclusions found in the various heats as a result of the radically different melting and solidification practices, it was difficult to correlate inclusions with solidification variables. However, in one sample, partially solidified at 1.15°F per minute and then water-quenched, substantial difference was obtained when comparing inclusions in the portion of the casting that was solidified slowly with those in the portion solidified rapidly. As would be expected, the more rapid cooling rate resulted in substantially finer inclusions.

C. Homogenization of Casting Structures

From the foregoing experimental results, it is clear that dendritic grain structure and dendritic segregation persist over the range of cooling rates studied (from 1.0°F per minute to substantially greater at 750°F per minute). Because of the probable importance of dendritic segregation (micro-segregation) in affecting mechanical properties, it was felt to be important to consider at this point in the investigation (1) the difficulties inherent in homogenizing cast ferrous alloys, and (2) the effect of dendrite arm spacing (and hence cooling rate and/or alloy analysis) on the ability to achieve homogenization at reasonable times and temperatures.

A simplified model of dendritic structure has proved to be of considerable usefulness in analyzing effects of thermal treatments on degree of homogenization. In this model, concentration gradients across dendrite arms and interdendritic areas are considered to be sinusoidal (Figure 8), and dendrite arms are assumed to be plate-like; i.e., diffusion is one-directional.

With this model, the initial concentration of segregated solute C_i is:

$$C_i = C_o + (C_m - C_o) \sin \frac{X}{\ell} \qquad (1)$$

Where: C_i = initial concentration at any point along the X axis (see Figure 8).

C_o = nominal (average) composition of the alloy.

C_m = initial maximum solute concentration.

X = distance along axis (see Figure 8).

ℓ = one half the dendrite arm spacing.

After a time θ, at a temperature T, the concentration profile is smoother and can be expressed mathematically by:

$$C_\theta = C_o + (C_m - C_o) \sin \frac{X}{\ell} \times e^{-\frac{\pi^2 D\theta}{\ell 2}} \qquad (2)$$

Where: C_θ = concentration at X at time θ

θ = time

D = diffusion coefficient at temperature T

The extent of homogenization taking place at any temperature at the time stated can be simply expressed by:

$$\delta = \frac{C'_m - C_o}{C_m - C_o} \qquad (3)$$

Where: δ = "degree of homogenization"

C'_m = maximum solute concentration at time θ

Note that δ is a direct measure of the change in composition of interdendritic areas with time. Before <u>any</u> homogenization has taken place $C'_m = C_m$ and $\delta = 1$. When homogenization is complete, $C'_m = C_o$ and $\delta = 0$.

Combining equations (2) and (3), δ can be related to diffusion coefficient, time at temperature and dendrite arm spacing:

$$\delta = e^{-\frac{\pi^2 D\theta}{\ell^2}}$$

Note the "degree of homogenization", as expressed above, is independent of initial solute concentration (provided the diffusion coefficient is independent of concentration). Degree of homogenization, δ , is plotted in Figure 9 versus homogenization time for several experimental conditions. Temperature in each case is 2200°F. Values and sources of diffusion coefficients used in this and subsequent calculations are given in Table II.

In the case of homogenization of nickel in a cast structure with a dendrite arm spacing of 300 microns, essentially <u>no</u> homogenization takes place until over 3 hours at 2200°F, Figure 9. More than 200 hours are required for the initial concentration differences to be reduced by 90 percent. Commercial homogenization treatments for low alloy steel seldom exceed 2200°F and are generally for only a few hours. Clearly, such treatments are not adequate to homogenize nickel in a cast alloy of this dendrite arm spacing.

Yet a dendrite arm spacing of 300 microns is not unusually coarse. Much coarser spacing is found in large castings and ingots, and even in ectionally solidified ingots. In a unidirectional ingot, for example, es away from the chill, dendrite arm spacing is in the order of 300 microns. Small experimental ingots studied in a recent investigation by Colling, Ahearn, and Flemings[10] had dendrite arm spacings of approximately 300 microns in regions examined by the electron microprobe. This study showed, experimentally, that essentially <u>no</u> homogenization took place (of elements such as nickel) after 2 hours at 2200°F; this is to be expected from the above calculations.

Times required for homogenization at a given temperature can be reduced significantly if dendrite arm spacings are reduced. Such reductions can be accomplished through chemical means or by increasing cooling rate.* For example, in this study (Figure 1) it was shown that alteration of chemical analysis decreased dendrite arm spacing by a factor greater than 10 at the slower cooling rates; it was also shown increasing cooling rate exerts an even more pronounced effect on dendrite arm spacing.

In steel of AISI 4340 composition, dendrite arm spacing next to a chill is the order of 30 microns in even relatively heavy castings. In ultra-thin section chilled steel castings, it may be as low as 10 microns.[36] Figure 9 shows the markediy faster homogenization time for nickel as dendrite arm spacing is reduced from 1,000 to 300, 30 and 10 microns; for 90 percent reduction in degree of segregation, the homogenization times required are, respectively, 2280, 206, 2.06 and .23 hours.

Diffusion coefficients for manganese and chromium (at homogenizing temperatures) are not greatly different from nickel and hence general conclusions derived above for nickel apply as well to these elements. Specifically, commercial heat treatment employed for low alloy steels should result in no significant solute redistribution of these elements (except in rapidly solidified material).

Homogenization of silicon occurs relatively more rapidly than the above elements, but the element affected most markedly is carbon, because

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* Alternatively the effective dendrite arm spacing (spacing of concentration gradations) can be reduced by mechanical working.

of its vastly higher rate of diffusion. Calculations indicate homogenization of carbon goes essentially to completion at 2200°F in very short times, even when dendrite spacing is relatively coarse.

Hence, although homogenization treatments do not, in practice, achieve anything approaching complete homogeneity of all elements, the foregoing calculations indicate they do have measurable and important effects on microsegregation of certain elements. Table III lists some comparative times required to achieve homogenization of various elements in steel (at 2200°F).

III. PLANNED WORK

Experimental and analytical work during the first 6 months of this research program has been largely exploratory in nature. A new technique for studying solidification of steel alloys was developed that makes it possible to examine effects of cooling rate and other variables on structure and segregation, without pouring large castings or ingots. Analytical studies were conducted that indicate that little or no homogenization of major alloy elements (other than carbon) occurs in heat treatment of low alloy steel ingots and castings.

Because of success obtained in the preliminary solidification experiments, improved apparatus was acquired to permit studying directly alloys of higher melting points (such as low alloy steels). A controlled atmosphere industrial furnace was obtained, and a programming temperature controller assembled, Figure 10. Figure 11 is a schematic illustration of the apparatus. The furnace and controller will permit solidification under controlled atmosphere at rates ranging from approximately 500°F per minute to less than 0.005°F per minute. This latter, slow, cooling rate is equivalent to a solidification time in excess of 10 days.

Future work planned includes studies using this apparatus to investigate in detail the effects of cooling rate on structure and segregation in cast steel. Exploratory work already conducted on techniques for measuring microsegregation (electron microprobe, autoradiography, etc.) will be extended to these carefully controlled experiments.

Planned work also includes a study on solidification and structure in small plate castings. Primary aims are to study and control solidification (1) under conditions where microporosity can be expected to be influenced by experimental variables, and (2) in a casting large enough so that test bars can be cut and mechanical properties related to solidification. (Mechanical property determinations are anticipated only when time and funds permit.)

Figures 12 and 13 show the type test to be used. Plate castings, 3" x 4" x 1/4" thick, are shell investment cast; each mold contains two plates. Techniques will be employed, using the plate casting, to vary cooling rate and other solidification conditions (and perhaps vary subsequent thermal treatments). It is then planned to examine in detail effects of these variables on structure, segregation, and, eventually, properties.

IV. CONCLUSIONS

1. Decreasing cooling rate results in increased dendrite arm spacing in low alloy steel. This is true in unidirectional, columnar solidification and in equiaxed, non-directional solidification.
2. Dendrite arm spacings up to 900 microns were obtained in a iron-nickel-carbon alloy solidified at 0.43°F per minute. Smaller spacings were obtained at faster rates of cooling.
3. Alloy analysis was found to have a marked influence on dendrite arm spacing. A steel containing approximately 25% nickel, 15% chromium, had substantially finer dendrite arm spacing (at a given cooling rate) than the less highly alloyed steels studied; for example, at a cooling rate of 1°F per minute, dendrite arm spacing in the nickel-chromium-iron steel was 84 microns. In AISI 4340 at a comparable cooling rate it is approximately 600 microns.
4. Dendrite arm spacing in the nickel-chromium-iron alloy was much less sensitive to cooling rate than was spacing in the low alloy steels.
5. In studies on solidification at relatively slow rates, controlled atmosphere is essential to avoid excess oxidation.
6. Decreasing cooling rate results in coarser, more unevenly distributed inclusions.
7. Dendrite arm spacing is the primary structural factor in castings and ingots affecting efficiency of homogenization (solutionizing) heat treatments.

8. Relatively coarse dendrite arm spacings are obtained in castings (particularly moderately heavy section castings) and ingots. Based on these spacings, calculations for low alloy steel indicate that:
   (a) Essentially no homogenization of alloy elements such as Ni, Cr, Mo, results from typical commercial homogenizing treatments (e.g., 2 hours at 2200°F).
   (b) Some homogenization of silicon generally occurs.
   (c) Essentially complete homogenization of carbon occurs (assuming carbide decomposition occurs readily).
9. Rapid cooling rates result in a fine dendritic structure. The structure in cast low alloy steel solidified in thin section chilled molds is sufficiently fine that it should be practical to obtain essentially complete homogenization in reasonable times at 2200°F.
10. The marked effect of alloy elements on dendrite arm spacing indicates that substantial reductions in spacing might be obtained by controlled additions to commercial alloys. Through such reduction, effective homogenization treatments could become possible, and with them improved mechanical properties.

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TABLE I

EFFECT OF COOLING RATE ON SECONDARY ARM SPACING

Cooling Rate °F/min.	$(\text{Cooling Rate})^{-1/2}$ $(°F/min)^{-1/2}$	Dendrite Arm Spacing (Microns)
Water quench	0.0	26
720.0	.037	48
75.0	.115	52
1.15	.93	70
1.0	1.00	84

TABLE II

DATA EMPLOYED FOR HOMOGENIZATION CALCULATIONS

$$D = D_o \times e^{-\frac{Q}{RT}}$$

where: D_o = pre-exponential factor (cm^2/sec)

Q = activation energy for diffusion (calories/gram)

R = gas constant (1.99 cal/°K mole)

T = absolute temperature (°K)

Element	D_o	Q	D (at 2200°F)	Reference
Mn (calculated for Mn = .80% C = .40%)	-	-	1.57×10^{-10}	(38a)
Ni (calculated for Ni = 1.8% C = .40%)	-	-	7.03×10^{-11}	(38b)
Cr (interpolated)	-	-	2.5×10^{-9}	(38c)
Si (extrapolated)	-	-	1.68×10^{-8}	(38d)
C (calculated)	.12 ± .07	32,000 ± 1,000	2.23×10^{-6}	(39a)

TABLE III

TIME REQUIRED FOR HOMOGENIZATION OF ELEMENTS IN STEEL (at 2200°F)

Times listed are those required at a given dendrite arm spacing to reduce concentration difference by 90%, i.e., $\delta = 0.1$.

Element	Time for Homogenization (Hours)			
	d = 1000 (micron)*	d = 300 (micron)	d = 30 (micron)	d = 10 (micron)
Mn	1020	92	.92	.10
Ni	2280	206	2.06	.23
Cr	63.8	5.75	5.75×10^{-2}	6.4×10^{-3}
Si	9.5	.855	8.55×10^{-3}	9.5×10^{-4}
C	7.2×10^{-2}	6.45×10^{-3}	6.45×10^{-5}	7.2×10^{-6}

* d = dendrite arm spacing, microns ($d = 2\ell$).

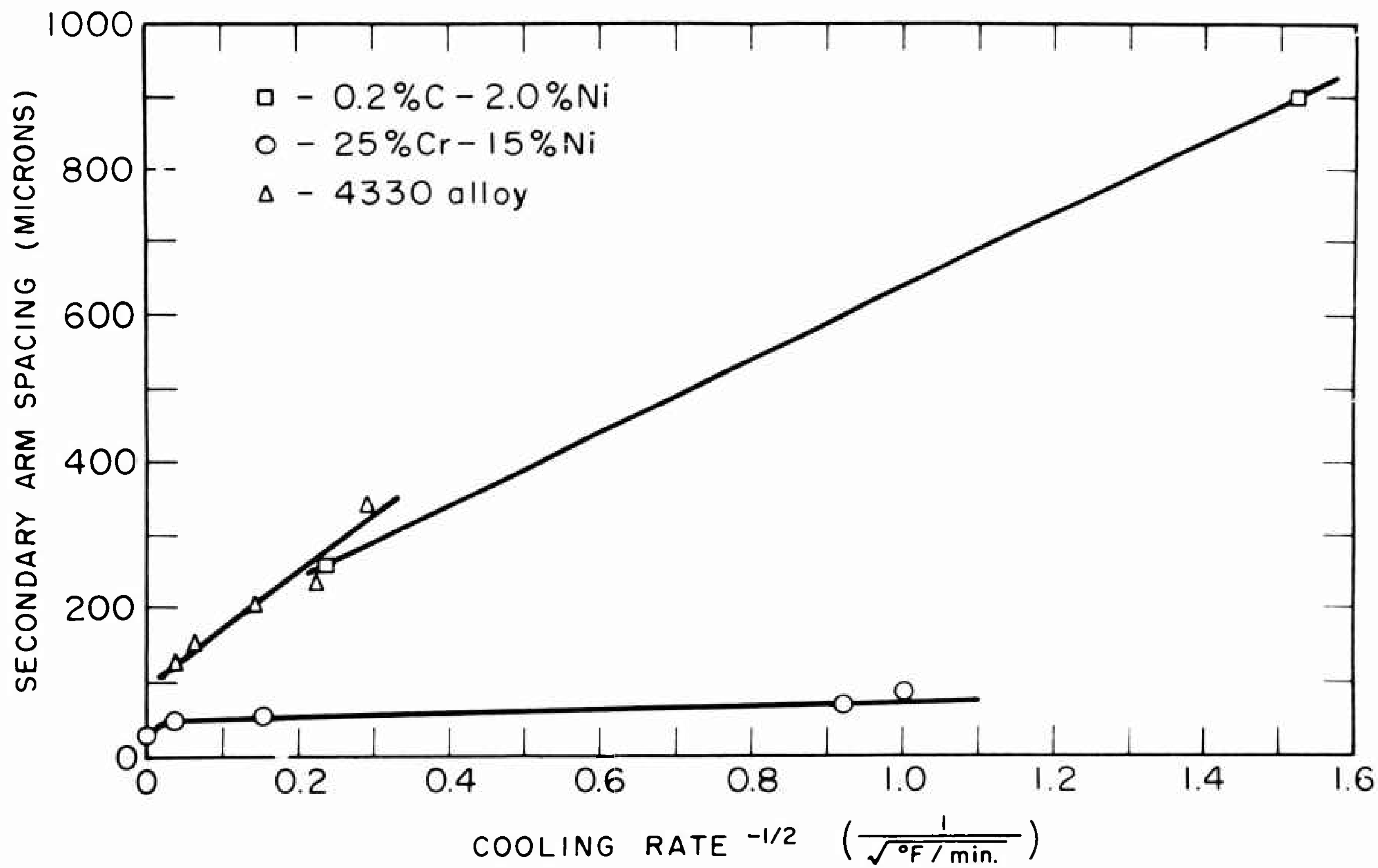

Figure 1: Dendrite arm spacing versus reciprocal of the square root of cooling rate.

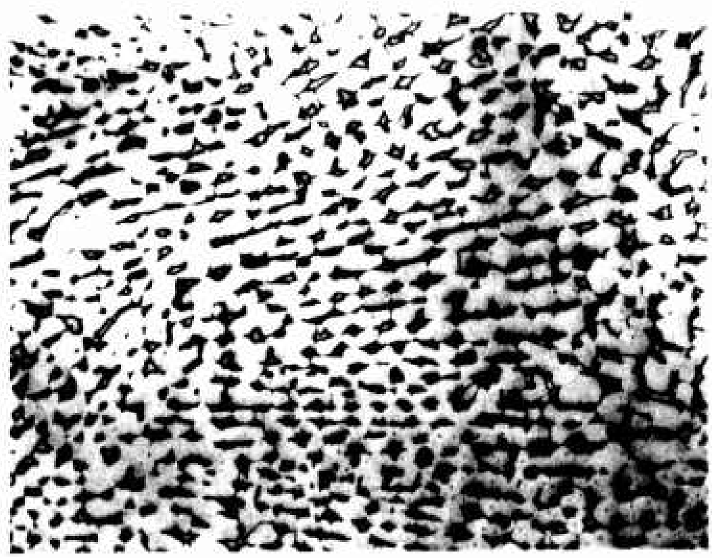

Figure 2. Dendritic structure of Fe-Ni-Cr alloy, cooled to just below the liquidus and water quenched. 80X.

Figure 3. Photomicrograph showing, on the left, a dendrite formed during slow solidification. On the right is the fine dendrite structure resulting from water quenching the partially solid sample. 1500X.

Figure 4. Dendritic structure of Fe-Ni-Cr alloy, cooled at 720°F. per minute. 12X.

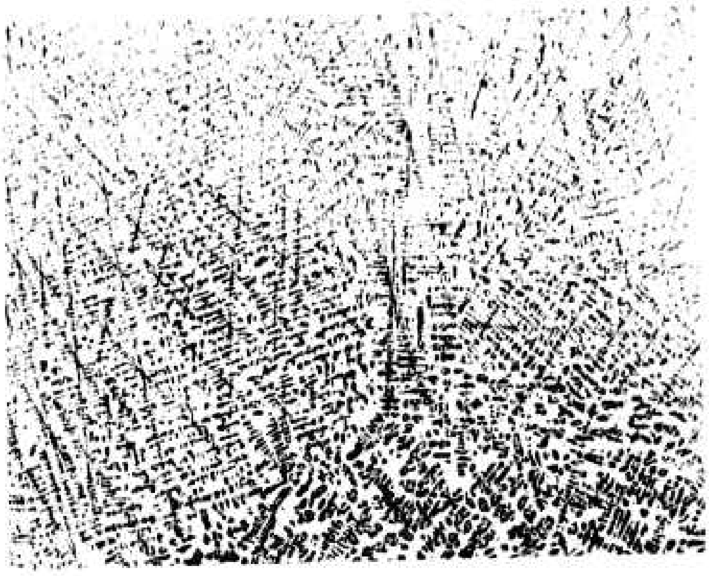

Figure 5. Dendritic structure of Fe-Ni-Cr alloy cooled at 75°F. per minute. Sample water quenched just below solidus. 12X.

Figure 6. Dendritic structure of Fe-Ni-Cr alloy cooled at 1.15°F. per minute. Water quenched just before complete solidification. 12X.

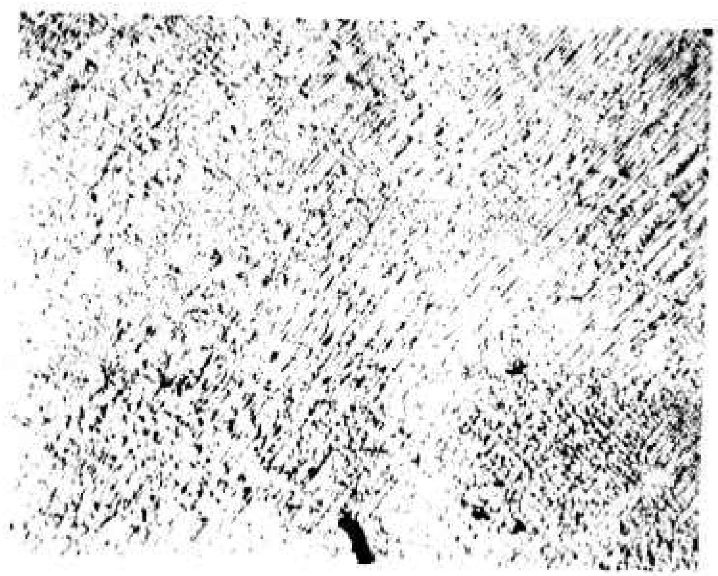

Figure 7. Dendritic structure of Fe-Ni-Cr alloy cooled at 1°F. per minute. Water quenched just below solidus. 12X.

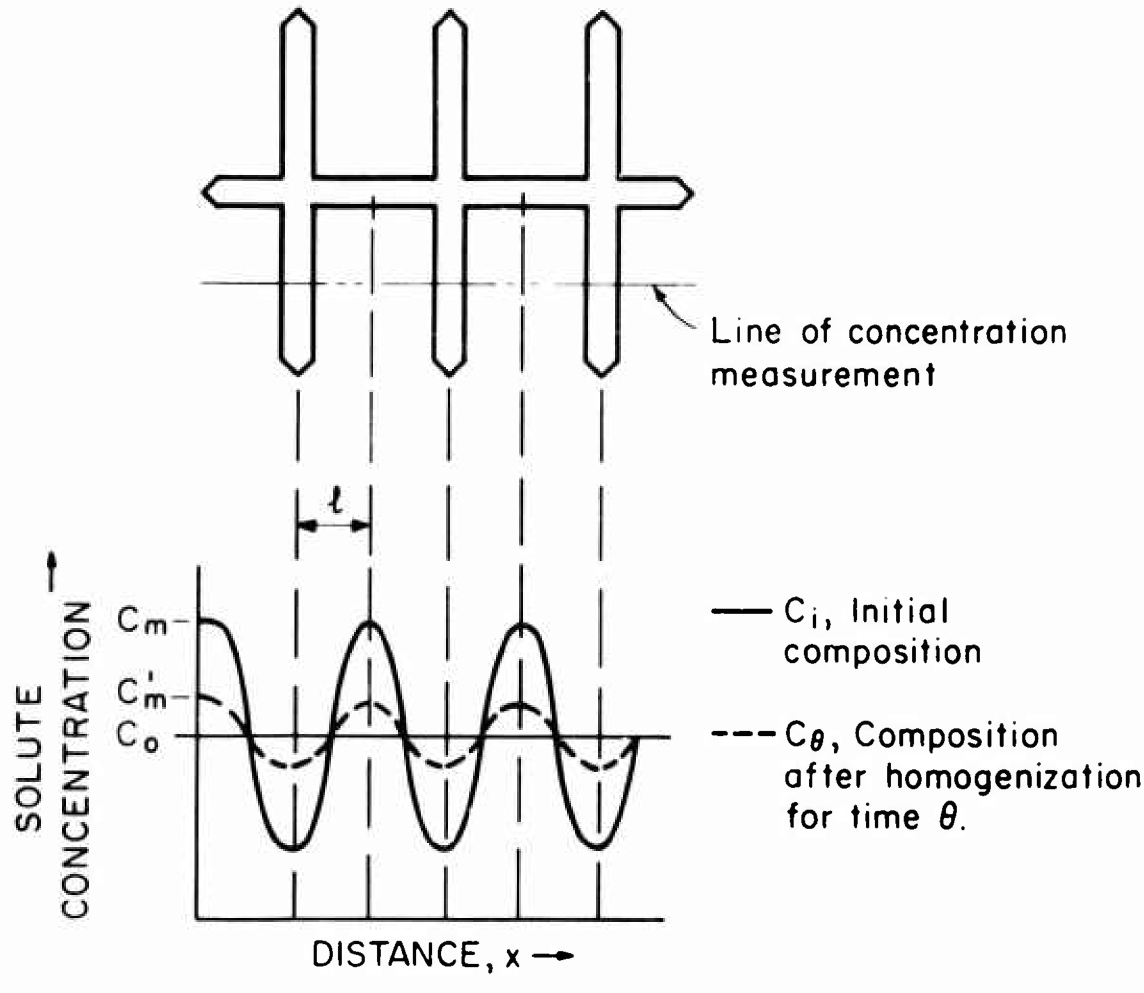

Figure 8: Schematic diagram of model of dendrite and dendritic segregation.

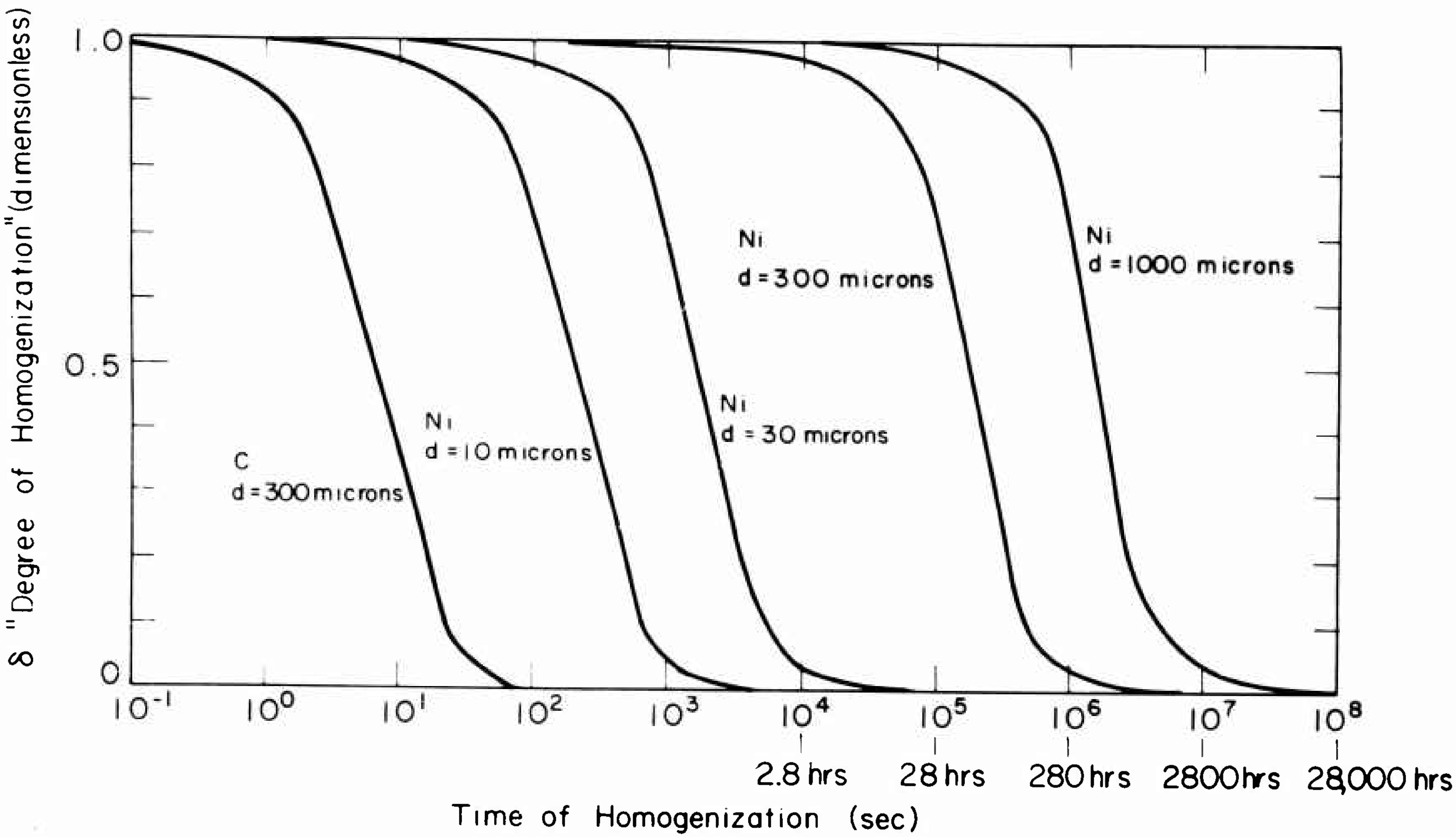

Figure 9: "Degree of Homogenization" versus time at 2200°F for nickel and carbon. (d = dendrite arm spacing)

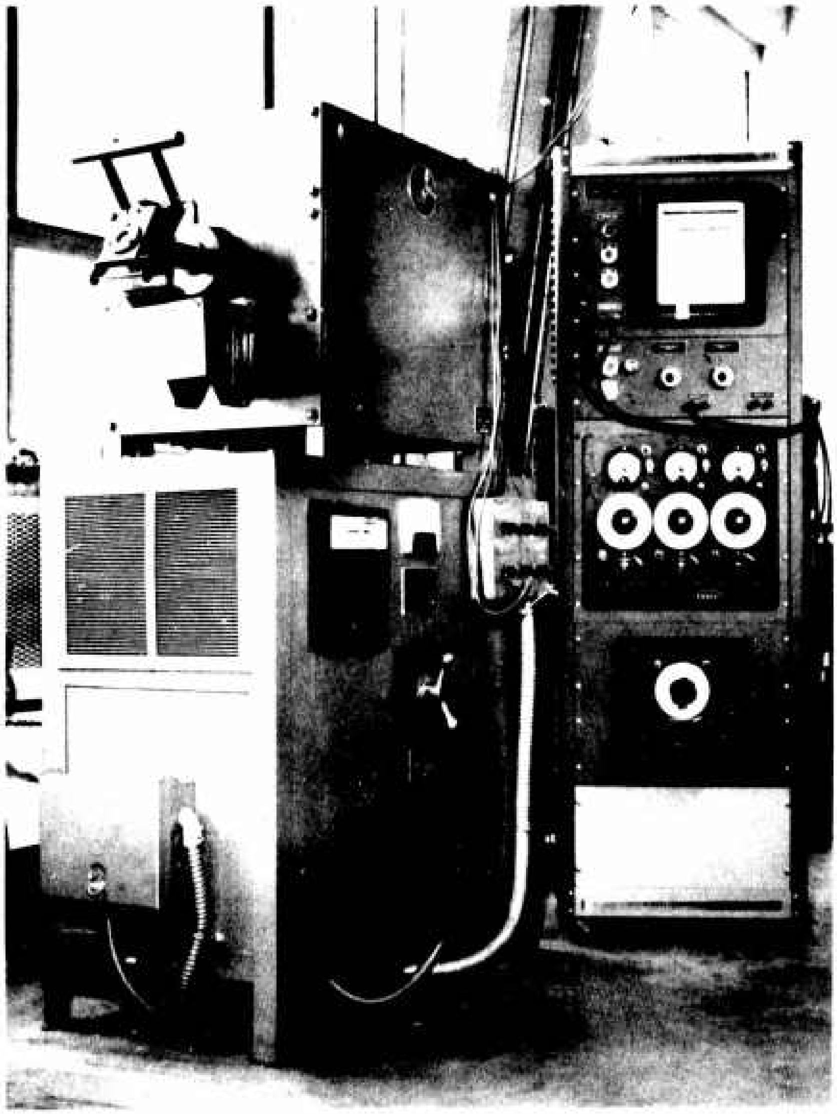

Figure 10. Photograph of recently installed furnace and programming temperature control unit for controlled solidification studies.

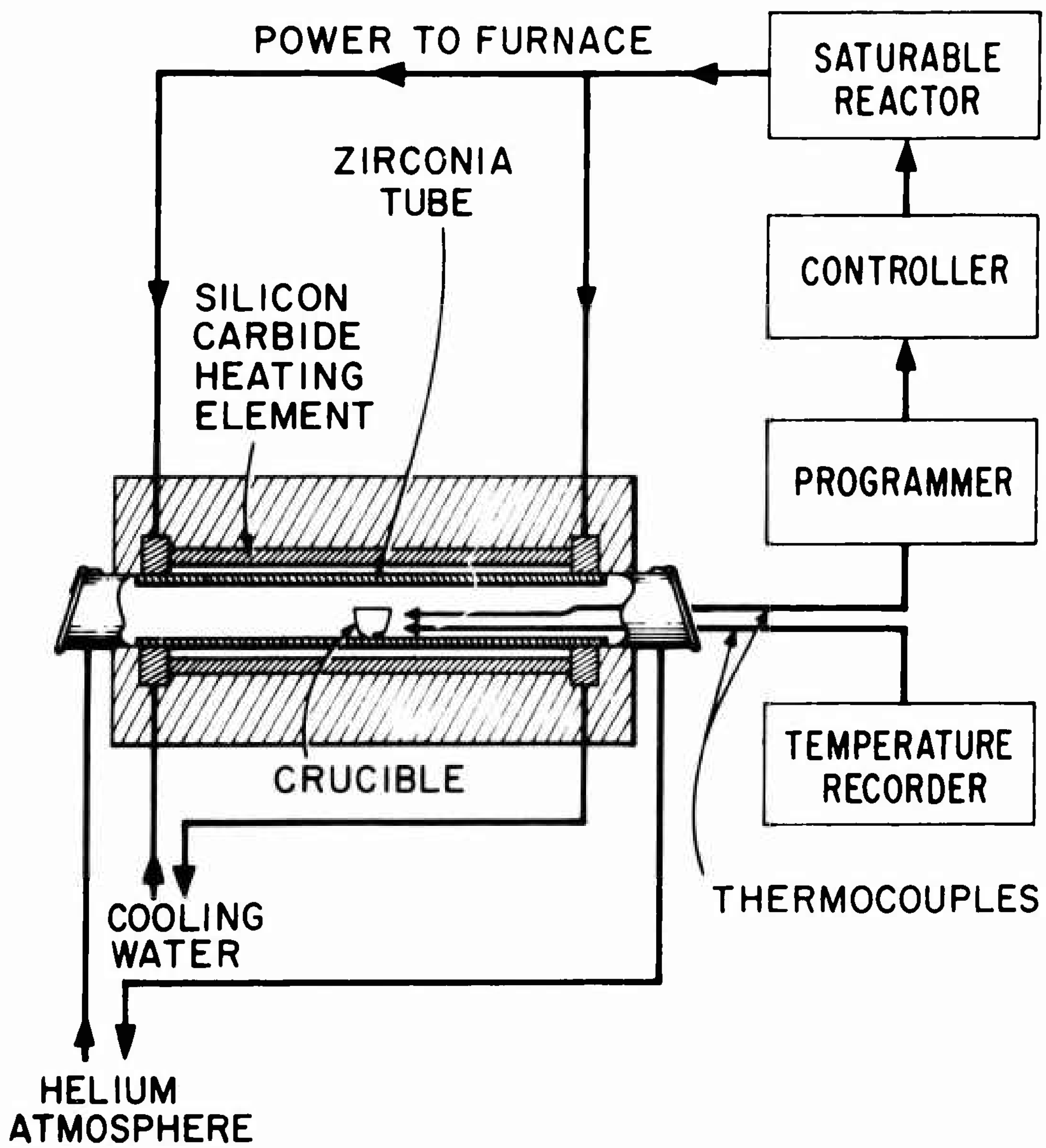

Figure 11: Sketch of apparatus for controlled solidification studies.

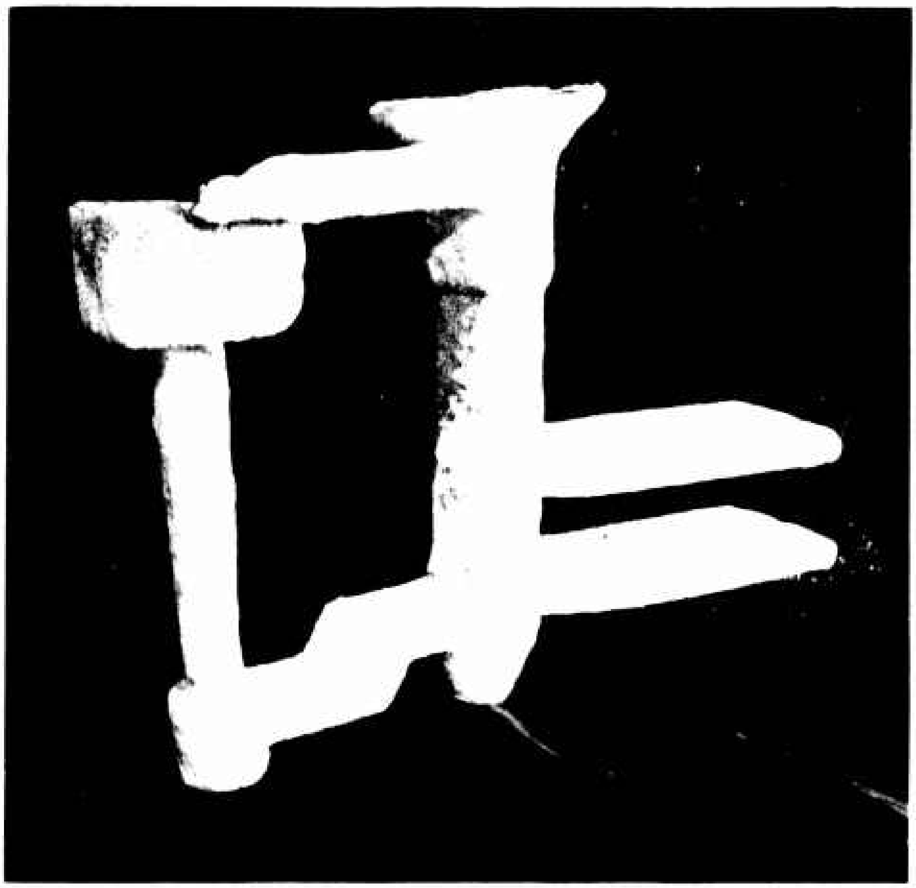

Figure 12. Photograph of shell mold for pouring plate castings. Two plates are poured in each mold; they are 4" x 3" x 1/4".

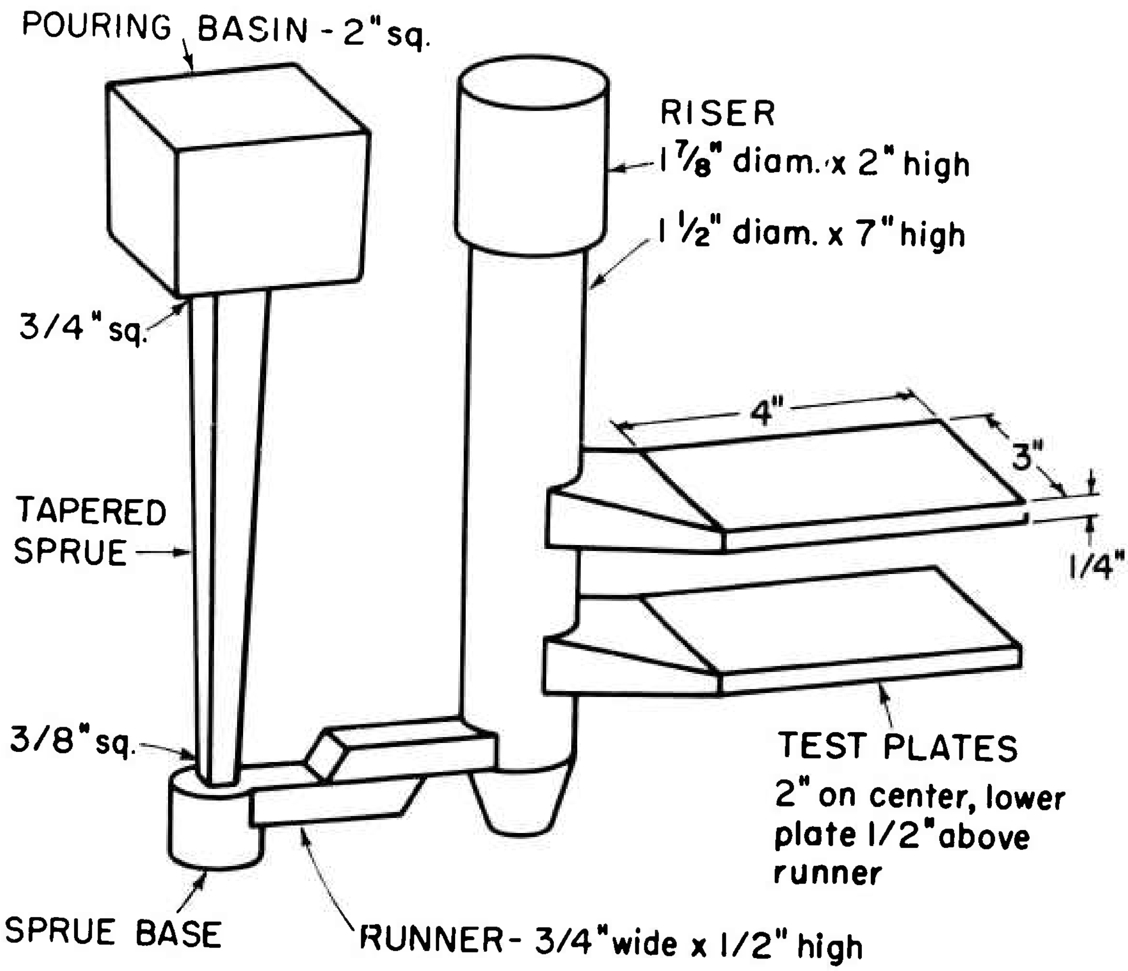

Figure 13: Sketch of plate casting and rigging system employed.

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